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SYNTHESIS OF $FeAl_2O_4$ UNDER A SHOCK-WAVE EFFECT

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$FeAl_2O_4$ is synthesized under the effect of shock-wave impact on gibbsite placed in a steel preservation ampoule. The type and parameters of the unit cell of this compound are determined. The x-ray patterns of the condensed products obtained on shock-wave impact are presented.

The pulse effect of strong shock waves (front pressure of 10 – 80 GPa, impact time of $2 \times 10^{-6} - 5 \times 10^{-6}$ sec) is used for the purpose of modifying the crystal structure of the known chemicals: carbon, boron nitride, silicon oxide [1 – 4], etc. Material samples suitable for further research and industrial processing are obtained in special devices known as preservation ampoules, which are divided into three groups [5 – 7] (Fig. 1) by the principle of the shock-wave action: flat (up to 5 GPa), axisymmetric (up to 80 GPa) and spherical (up to 1000 GPa) actions.

The volume of compounds condensed in compression usually varies within the limit of 20 – 30% and is compensated by deformation of the preservation ampoule. In the present work, an attempt was made to preserve the products of decomposition of gibbsite (analytically pure grade, GOST 11841–66) produced by shock-wave impact. These products,. apart from condensed oxides, contain a substantial amount of water which generated steam in the experiment and, as a consequence, caused destruction of the ampoule of standard design (Fig. 2*a*). An unusual phenomenon is observed in this case: the chemical reaction which is endothermic by nature ($\Delta H_p = 38.7$ Kcal/mole),

$$Al_2O_3 \cdot 2H_2O_{(k)} \rightarrow Al_2O_{3(k)} + 3H_2O_{(g)},$$

under the conditions of shock-wave impact is accompanied by an explosion.

In this context, the design of the preservation ampoule was modified. The device was designed as a steel waveguide rod 13 mm in diameter; the sample (0.04 g) of the tested ma-

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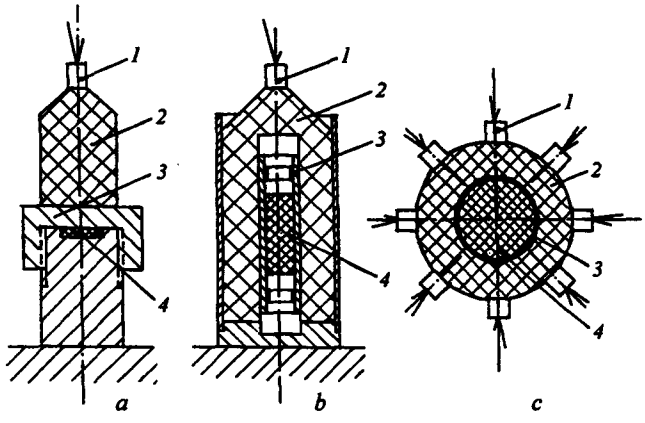

Fig. 1. Design of experimetal devices used for synthesis of new crystal structures under the effect of flat (*a*), axisymmetric (*b*) and converging spherical (*c*) shock-wave impact. *1* — Detonation initiator; *2* — explosive charge; *3* — preservation ampoule; *4* — tested sample.

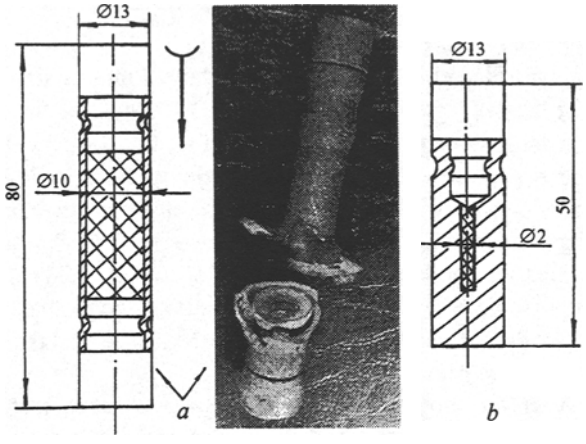

Fig. 2. Design of the preservation ampoule for axisymmetric loading of a sample, view after the experiment involving an explosion (*a*), and the improved design (*b*) intended for camouflet explosion. The arrow indicates the direction of propagation of the detonation wave in the charge.

TABLE 1

Intensity	Interplanar distances D_e, E	Miller indexes *hkl*
33	3.4695	012*
58	2.8785	112
48	2.5476	104*
100	2.4538	103
23	2.3744	110*
48	2.0820	113*
11	2.0281	004
23	1.7390	024*
11	1.6623	132
48	1.6006	116*
28	1.5662	033
33	1.4385	224

* Corundum lines.

TABLE 2

Intensity	Interplanar distances D_e, E	$Q = 10^4/D_e^2$	*hkl*	$Q = 10^4/D_e^2$	*dQ*
58	2.8785	1206.92	020	1207.0	−0.1
58	2.8785	1206.92	112	1209.2	−2.2
100	2.4538	1660.80	013	1664.4	−3.6
100	2.4538	1660.80	121	1660.2	0.6
11	2.0281	2431.31	004	2422.6	8.7
11	1.6623	3619.02	132	3623.2	−4.2
28	1.5662	4076.46	133	4078.5	−2.0
28	1.5662	4076.46	231	4074.3	2.2
33	1.4385	4832.51	040	4828.1	4.4
33	1.4385	4832.51	224	4836.6	−4.1

terial was placed inside a cylindrical hole 2 mm in diameter along the waveguide axis (Fig. 2*b*). The walls of the ampoule could withstand the explosion of $Al_2O_3 \cdot 3H_2O$ without being destroyed. The ampoule containing the sample was placed coaxially inside a 36.5 mm diameter charge of octogen surrounded by a shell with a wall thickness of 4.5 mm. The bulk density of the octogen was 1.06 g/cm^3. The ampoule is exposed to a gliding detonation wave. The octogen detonates with a velocity of 6.2 km/sec, and the detonation wave pressure is 12.2 GPa.

After the ampoule was opened, it was found that the transformation products had been compacted and were deposited in the form of a ring on the walls of a through channel 1 mm in diameter formed along the axis of the ampoule.

The x-ray phase analysis of the preserved material was performed in a FR-555 focusing monochromator chamber ($CuK\alpha_1$-radiation, germanium as a referencce standard).

The x-ray pattern (Fig. 1), apart from six clear lines of corundum with *hkl* 012, 104, 110, 113, 024, and 116, exhibited six supplementary lines as well. The x-ray data (relative intensities and interplanar distances) corresponded to the phase of $FeAl_2O_4$ (ASTM 34–192). In contrast to the published data, the x-ray pattern of the $FeAl_2O_4$ phase obtained by us was indexed in a body-centered tetragonal cell: $a = 5.7567\ (34)$ E, $c = 8.1269\ (98)$ E, $V = 269.32\ (03)$ E^3. The results of indexing of the x-ray pattern of the $FeAl_2O_4$ phase produced by shock-wave impact on aluminum hydroxide in the steel ampoule are given in Table 2.

Earlier [8], $FeAl_2O_4$ was synthesized from a stoichiometric mixture of thoroughly pulverized components (α-Fe_2O_3 and γ-Al_2O_3) in a steel capsule with excessive oxygen pressure for 24 h with periodic heating in the temperature interval of 1200 – 1300°C.

The extremely high speed of $FeAl_2O_4$ synthesis under shock-wave impact complicated by the initial heterogeneity of the experimental conditions probably ensues from the possibility of attaining high temperature (the melting point of $FeAl_2O_4$ is 2135°C), dynamic mixing of the reactant phases in circumstances of complex hydrodynamic flow, and transition of the condensed compound into a specific state in the pressure range around 30 GPa [9].

Thus, the possibility of synthesis of spinel under the effect of shock-wave impact was demonstrated.

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